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DESIGN AND TEST OF POROUS-TUNGSTEN MERCURY VAPORIZERS

by William R. Kerslake
Lewis Research Center
Cleveland, Ohio

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William R. Kerslake
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

Abstract

Future use of large size Kaufman thrusters and thruster arrays will impose new design requirements for porous plug type vaporizers. Larger flow rate coupled with smaller pores to prevent liquid intrusion will be desired. This paper presents the results of testing samples of porous tungsten for flow rate, liquid intrusion pressure level, and mechanical strength. Nitrogen gas was used in addition to mercury flow for approximate calibration. Liquid intrusion pressure levels will require that flight thruster systems with long feed lines have some way (a valve) to restrict dynamic line pressures during launch.

Introduction

Porous plug vaporizers have been successfully used to supply vapor mercury propellant to both ground-based research tests and flight operation of Kaufman thrusters for the past eight years.[1-3] Porous tungsten has been chosen to date as the best material because of its excellent corrosion resistance to mercury and because of a large material technology base existing from its past use in cesium contact ion thrusters.[4] A suitable porous material must have, (1) a liquid intrusion pressure higher than the operating vapor pressure, (2) material strength and workability suitable for fabrication into feed systems, (3) negligible corrosion after tens of thousand hours of use, and (4) be nonwetting with liquid mercury.

Existing porous tungsten fulfills these requirements, but future space missions will have additional requirements. Because arrays of thrusters will be used, feed lines will be used between a common reservoir and each thruster. During launch vibrations dynamic pressures of 100 to 1000 psi may exist in these lines. The pores must be small enough to stop liquid intrusion at these pressures, or high pressure mercury cannot be permitted in these lines during launch. Future thrusters will also operate at much higher flow rates. Greater flow rate through small pores requires, (a) a large diameter vaporizer (which in addition to using more heating power, has in the past caused welding fabrication problems), (b) a thinner porous plug (with subsequent unknown stress-rupture limits), or (c) vaporizer operation at higher temperature and pressure levels (with corresponding penalties for a higher pressure propellant supply system).

Attempts to improve flow rate and reduce pore size were made by fabricating samples using different powder sizes of tungsten and a range of sintering temperatures and times. The samples of porous tungsten thus produced were tested for vapor flow rate, liquid intrusion pressure level and mechanical strength. These results together with a design section which enables the results to be applied to future thruster systems are presented in this paper.

Design Theory

General Vaporizer Operation

A cross section of a typical flight thruster vaporizer is shown in Fig. 1(a). Figure 1(b) shows the configuration used to test the experimental samples of this paper. Liquid mercury is supplied under pressure from a reservoir or tank. This flow is stopped by a circular disk of porous tungsten welded into the tube. Liquid mercury does not enter the porous plug because mercury does not normally wet tungsten, nor is the liquid supply pressure high enough to force the liquid, against its surface tension, into the pores.

The heater raises the liquid mercury and walls to a temperature level of 300° C. (A slip-on heater was used for the experimental configuration.) Boiling does not occur because the liquid supply pressure is several times greater than the equilibrium vapor pressure at 300° C. The equilibrium vapor, about 3.3 N/cm^2 (250 torr), is free to leave the liquid surface at the pore openings of the plug. The vapor flow rate is determined by the porosity of the plug and the temperature (vapor pressure) of the liquid. The liquid pressure does not influence the vapor flow, but merely serves to keep the liquid interface against the plug. Typically, beam current or discharge voltage is sensed and used in a closed loop control of the vaporizer heater current.

Vapor Flow

As porous tungsten is manufactured in batches, a sample can easily be checked for flow conductance over a range of interest and the following method used to extrapolate this flow measurement to other flow rates, gases, or sizes of vaporizer. (The rate of vapor flow through a porous media may also be calculated by assuming flow takes place through a bundle of small, parallel capillaries.[5-7] But this method depends on generally unknown physical material properties and will not be used herein.)

The measured sample flow is normalized by computing the ratio of the number of gas molecules leaving the downstream plug surface to the number arriving at the upstream surface. This ratio C is called the transmission coefficient. Its value for a porous thickness ℓ of 0.1 cm is typically 10^{-4} to 10^{-5}. The value of C can be made larger or smaller by changing the number or size of the pores. The value of C is also inversely proportional to ℓ, the plug thickness. Knowing C, the following equation can be used to calculate flow rate for other gases and over a range of temperature and pressure.

$$j_o = C\left(e\sqrt{\frac{N_o}{2\pi k}}\right)\frac{P_v}{\sqrt{T_v M}}$$

or

$$j_o = C \frac{0.422 \times 10^6 \, P_v}{\sqrt{T_v m}} \qquad (1)$$

where

j_o gas flow leaving plug, equivalent A/cm^2

C transmission coefficient

P_v upstream vapor pressure, N/cm^2

T_v the upstream vapor temperature, ^oK

e 6.24×10^{18} atoms/sec per equivalent ampere of flow

M gas molecular weight, amu

N_o Avogadro number, 6.02×10^{26} atoms/kg mole

k Boltzmann constant, 1.3805×10^{-23} joules/^oK

The right side of Eq. (1), with C removed, is the molecular arrival rate at a surface in units of equivalent A/cm^2. (Each atom or molecule is assumed to have the charge of one electron.)

The value of C depends only upon the porous tungsten properties and is independent of P_v, T_v, or M as long as flow in the pores is free molecular. As will be shown in the results, C is constant for upstream pressures up to about 1.3 N/cm^2 (100 torr) for the samples tested. Above this pressure, C begins to increase as the flow in the pores goes into slip or transitional flow. The break from free molecular flow to slip flow occurs as expected when the molecular mean free path aproximately equals the pore size which is about 2 microns diameter.[7]

Liquid Flow

If liquid enters the pores, the mercury flow is no longer controlled by the plug temperature, but depends on the liquid pressure and the wall friction of the pores. Under these conditions, the flow can be 100 times higher. The pressure at which a nonwetting liquid will enter a capillary is a function of the liquid surface tension, contact angle of wetting, and capillary diameter. The following equation gives an approximate relation between liquid mercury penetration pressure and capillary or pore diameter, for nonwetted (contact angle, 180°) surfaces. (The surface tension of mercury was taken to be 4.76×10^{-3} N/cm^2.)

$$P_p = \frac{190}{D_p}, \text{ pressure for penetration} \qquad (2)$$

where

P_p liquid intrusion pressure, N/cm^2

D_p pore diameter, microns

The highest liquid pressure experienced will in general occur during launch vibration and can be calculated by the following equation.

$$P_{VIB} = a \Delta L \rho \times 10^{-5} \qquad (3)$$

where

P_{VIB} vibration induced liquid pressure, N/cm^2

a vibration acceleration, cm/sec^2

ΔL length liquid head in direction of a, cm

ρ density of liquid, gm/cm^3

For a typical launch vehicle (Thorad) dynamic liquid pressures can be as high as 4 N/cm^2 (5.8 psi) per centimeter of propellant head.

During normal vaporizer operation the minimum liquid pressure must be 1 to 1.5 times the vapor pressure to insure that the liquid interface remains at the porous plug. Should the liquid recede and a vapor pocket form, the flow will be erratically reduced. The vapor pressure will be controlled by the receded interface which is at a lower temperature.

The maximum possible operating liquid pressure is the pore penetration pressure. Tankage weight and other penalties are usually associated with higher pressure operation, and operating liquid pressures near the minimum are desired for flight thruster feed systems. On the SERT (Space Electric Rocket Test) II flight, the design liquid pressure was 2 atm at full reservoir and 1 atm at empty reservoir.[8] The 1 atm empty pressure was about three times the minimum operating vapor pressure to provide reserve pressure in the event of tank leakage.

The above statements are valid if the mercury does not wet the porous material. This assumption is true for the general case of normally clean tungsten and mercury. Work done on SNAP 8 mercury boilers[9] and the wetting of metallic surfaces by mercury,[10] indicate that mercury will wet metallic surfaces under two conditions. One, the metallic surface must be ultra clean without even a monolayer of oxide present, or two, wetting agents or contaminates (dirt) must be present at the surface. Ultra clean tungsten can become covered with a monolayer of oxygen in 10^{-5} torr-seconds of exposure at room temperature[11] and such monolayers can be responsible for nonwetting by mercury. As doubt of the durability of this thin layer may exist, a thicker oxide layer, 10^3 angstroms thick[9] can be applied by gentle (200° C) heating of the porous tungsten in air. Caution should be used to avoid very thick films (>400° C) that might become loose during operating and expose a fresh unprotected tungsten surface. Gentle air heating was standard procedure for SERT II vaporizers. Incandescent temperatures are required to remove the normal oxide film from tungsten in a vacuum.

The surface must be kept free of metal contaminates to prevent mercury wetting. One documented[12] case indicated after 24 hours of vaporizer operation using mercury containing 1 percent silver impurity, that liquid mercury wetted and flowed through the porous vaporizer plug. Tests at the Lewis Research Center by J. F. Staggs and V. K. Rawlin indicated no wetting of porous tungsten after 300 hours of operation with copper intentionally plated on both surfaces of the vaporizer. In other tests up to 300 hours with various metal (copper, silver, zinc, gold, lead) immersed in the

liquid mercury immediately upstream of the porous plug did not cause wetting.

Considerable uncertainty therefore exists as to the maximum level and type of impurity that will prevent mercury-tungsten wetting. A safe level of impurities has been established by trouble-free operation during 50 000 hours of accumulative testing in the SERT II project. Feed system tests included seven tests of 1000 to 3000 hours, and two tests of 5000 hours. Many semiquantitative spectrographic analyses were made of the recleaned, triple-distilled mercury used in the SERT II tests. Random trace amounts of silver, gold, calcium, silicon, magnesium, and other metals were at times detected at levels of several parts per million or less. This level of impurity concentration, based on lack of observed wetting, is concluded to be safe for future flight thruster applications.

Mechanical Strength

In the future when smaller pore material is used to obtain higher liquid penetration thresholds, the value of C will decrease. To keep the same flow rate the porous material must be thinner.

The porous plug of the vaporizer must have sufficient strength, however, to withhold the liquid pressure without rupturing. The following equation was used to calculate the rupturing stress limit for the experimental vaporizer shown in Fig. 1(b). The porous plug was assumed to be a circular plate with a uniformly distributed load.[13]

$$S = \frac{k}{4} \frac{P_L D^2}{\ell^2} \qquad (4)$$

where

S rupture stress, N/cm^2

k a constant; either 1.24 for simply supported edge or 0.75 for a clamped edge.[13]

P_L liquid pressure force at rupture, N/cm^2

D diameter of plug, cm

ℓ thickness of plug, cm

Results presented later will show the value of S (assuming a k of 0.75) to be about 18 000 N/cm^2 (26 000 psi) at room temperature. To design a plug to operate at 300° C, a safety factor of about four should be used. This value includes a general safety factor of two and another factor of two to correct for a reduced stress rupture value at operating temperatures.

Other Porous Materials

As tungsten is not easy to fabricate, other porous materials have been considered. The SERT I thruster system used a porous stainless steel plug.[14] The RIT 10 (Radio frequency Ion Thruster, 10-cm diam) thruster uses a metal mesh.[15] The difficulty with stainless steel is a slight mercury corrosion, which by itself may remove negligible material, but this slight corrosion could cause an unknown change in the wetting properties. The usual difficulty with screens or meshes is an inability to obtain small enough openings to withhold liquid penetration.

A commercially available "collimated hole" filter material of stainless steel has possible use with a pore size of 13 microns. It is experimentally available in materials other than stainless steel. Collimated hole material has two advantages. First, the capillaries are smooth and parallel and would not trap or retain any accidentally intruded liquid mercury. Second, the material has low flow blockage and extremely high (50 equivalent A/cm^2) flows are possible. The disadvantages are a low liquid nitrusion pressure level due to relatively large capillaries, and not being commercially available in tungsten.

Most ceramics are nonwetting with mercury and many are available in a porous state. One unique advantage, should a ceramic be used as a vaporizer, is that it may also serve a dual role as an electrical isolator. (Present thruster systems require a ceramic portion of the vapor flow tube to provide electrical isolation between the thruster and the propellant reservoir.) The author has tried unsuccessfully to fabricate and test a porous ceramic plug within a nonporous ceramic flow tube. The difficulty has been in obtaining a porous ceramic with a high C value and yet with sufficient mechanical strength to prevent crumbling or crack formation at the bond to the nonporous ceramic wall.

The porous tungsten vaporizer, in spite of high cost and long fabrication time, presently is the first choice for future flight use. This is because of the past, trouble-free, successful application of porous tungsten and because of known, well-documented fabrication methods.

Fabrication

Porous Plug

The particular porous tungsten purchased and used by the Lewis Research Center was fabricated by a proprietory process, but several other manufacturers have made essentially similar quality porous tungsten. Tungsten is commercially available as powder. This powder is screened or classified to obtain a relatively uniform particle size of, say, 5 micron diameter. The powder is mechanically compressed, usually with some binder, to produce a desired slab or rod shape. The mechanically compressed sample is then sintered at high temperature to obtain partial bonding or fusion between particles.

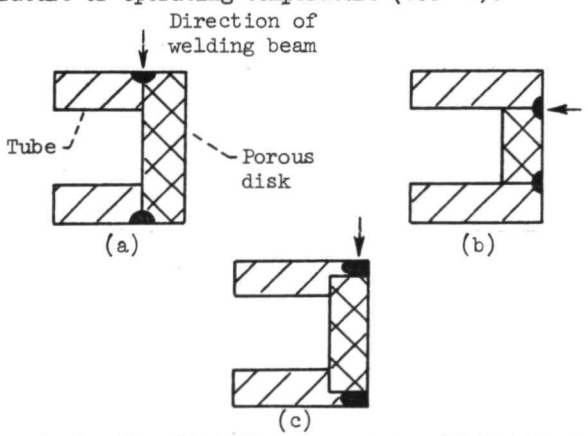
The porous tungsten used in the program was fabricated in slabs of pure tungsten, any binder being volatile and escaping during the sintering. Circular disks are cut by electric discharge machining (EDM) from slabs. As final plug thickness is the same as the slab thickness, both flow surfaces were original material and have no need for further processing. Machined surfaces usually have smeared material closing the pores and this must be removed by an etching technique.[6] After the EDM cut, a light cut is taken with a sharp tool to obtain a close tolerance with the counter bored tube in which the plug is welded. In addition, the edge of the plug is "washed" with an electron beam welder to insure closing of all edge pores and to prevent bypass vapor leakage.

Figure 2 shows electron-scan microphotographs of two finished plugs before welding into a tantalum tube. The magnification was X2000 with a 20° tilt. Sample 1 is seen to be lightly sintered with small areas of fusion. Sample 2, sintered at 250° C higher temperature shows a large amount of fusion between particles. The temperature level of sintering produced the most change in appearance between samples, with the time of sintering being a minor variable.

Assembly

Tantalum tubing was chosen to hold the porous tungsten because of a good match of coefficient of thermal expansion, lack of mercury corrosion, and availability of flaw-free tubing. Tungsten tubing was considered too brittle for mechanical support to mating components of the feed system. Molybdenum tubing, which has similar expansion and corrosion to tantalum, was rejected for lack of high-quality commercially available tubing.

As sketches below show, three ways were tried to weld the porous plug into the tube. Methods shown in sketches (a) and (b) were rejected because of large cracks that formed at the weld-porous material interface. The method shown in sketch (c) was the final way used, but some cracking still occurred. To avoid cracks, the plug and tube counterbore must be close fitted as possible. Also, the electron beam operator used a diffuse beam to warm up the parts to about 700° C before making the final weld. Using this method, the weld seam should be in compression rather than tension at either room temperature or operating temperature (300° C).



During the SERT II program the welded tube and plug were vacuum baked at 1850° C and 10^{-6} torr for 1 hour to remove any impurities that may be present. The chief impurity was copper that came from fixtures used to hold the tube and plug during electron beam welding. Subsequent vaporizers were made without this vacuum bake step and have operating in research programs without difficulty. This step may therefore be unnecessary for future flight thrusters.

After the vacuum bake, the welds were inspected for cracks. Cracks of width 50 to 200 microns could be observed with the help of a low power microscope. Smaller cracks could be detected most easily by the following procedure. A drop of a clean liquid (isooctane) was placed on the porous surface and gas gently flowed from the other side. If the assembly were crack free, small bubbles randomly appeared over the entire porous plug. If a crack were present, all the bubbles, larger in size, appeared over the crack. Final acceptance for a flight thruster included mercury vapor flow calibration and liquid penetration tests. Over 90 percent of the defective welds and cracks, however, were found by direct visual observation or the bubble test.

Electron-beam welding of tantalum to stainless steel is difficult because of the large difference in melting temperature. For this reason the tantalum flow tubes were extended both upstream and downstream. Upstream the tantalum tube was welded into a small tantalum flange which was bolted to a mating stainless steel flange and "O" ring seal on the mercury reservoir. Downstream the tantalum tube was welded into a tantalum cap, which was brazed over the end of a ceramic isolator tube. The entire flow system therefore was either welded or brazed together with the single exception of the mercury reservoir flange.

Experimental vaporizer plugs of larger diameters, 1.2- and 1.8-centimeter diameter were successfully welded for the present work. The present success is attributed to a better welding technique of preheating the tube before finally welding and possibly greater mechanical strength in the newer porous tungsten slabs. (Over 90 percent of previous plugs larger than 0.6-centimeter diameter could not be reliably welded into tantalum tubes.) There were no rejected welds in the four larger sample plugs prepared for this paper.

Heater Placement

The heater has been placed upstream, downstream, and directly over the porous plug. The preferred position is one in which a "direct" conduction heating path exists between the heater and the porous plug as shown in Fig. 1(a). If the heater were placed on the downstream tube, a variable heat path exists due to the region of overlap. The heat transfer between the outer and inner tube depends in part on the contact between tubes in this overlap region. Such heater placement caused flow oscillation in prototype vaporizers because as the heater controlled on and off, the relative expansion between tubes caused intermittent touching in the overlap region.

With a good thermal path to the porous plug and with the heater no more than one or two tube diameters upstream, the tube, mercury and porous plug is within 2° C for the area under the heater. If the heater were located 5 to 10 tube diameters upstream, a significantly lower temperature could exist at the porous plug. Vapor pockets could then form under the hotter heater area. Such vapor pockets would be unstable and may be displaced by liquid forces in either direction to a cooler region where they condense, causing pressure disturbances in the entire tube.

The heater itself is a tantalum wire swaged within a tantalum tube filled with alumina and then brazed on the vaporizer tube. The coils are slightly separated to insure braze flowing between and under all loops of the heater. Loose-fitting caps are placed over the ends of the vaporizer tube during heater brazing to retard the contact of any impurities in the brazing furnace with the porous plug surfaces.

Apparatus and Procedure

Mercury Flow Determination

Figure 3 shows a schematic representation of the apparatus used to test the porous samples for mercury vapor flow and liquid intrusion pressure level. The sample tube was placed inside a bell jar, which was pumped to a pressure of 10^{-4} torr or less. The mercury was metered and supplied to the test sample by means of a pressurized 0.051±0.001 cm precision bore glass capillary tube. Before filling the lines with liquid mercury, they were evacuated to 1×10^{-2} torr with a mechanical pump to remove air and prevent compression or expansion errors from residual gas. The mercury filled lines and capillary tube were thermally insulated to reduce mercury expansion and capillary tube reading errors due to room air temperature fluctuations. In addition, an ordinary thermometer was hung next to the apparatus and data was not taken if the rate of change of the room air temperature was more than 0.1° C per minute.

The sample was heated to the test range (280° to 370° C) and the feed system was allowed to stabilize for 1 hour or more. The feed system was pressurized with nitrogen to a total pressure (including liquid heat) of 17 N/cm^2 (25 psia). This pressure was necessary at the higher temperature test points to hold the liquid interface at the plug. The fall of mercury in the capillary was timed successively until a consistent set of three readings were obtained. This procedure was repeated to obtain mercury flow rates as a function of temperature over the test range. For each temperature the vapor pressure was obtained from a table in Ref. 16, and the transmission coefficient C was computed for that point.

At a single test temperature, the nitrogen pressure was increased stepwise to 51 N/cm^2 and any change in vapor flow measured. The vaporizer was then cooled to room temperature and the nitrogen pressure increased until the liquid mercury intrusion level was observed. The vapor flow was remeasured to observe any shift in C due to the mercury penetration. Next, the mercury was drained from the lines and the vaporizer was heated to 400° C for 15 minutes to bake out any residual mercury trapped in the pores. The lines were refilled and vapor flow was again measured. A repeat measurement of the liquid intrustion level was also done.

The reproducibility of mercury flow measurements was normally ±2 percent and with longer time intervals (2 hrs) of measurement was ±1 percent. The absolute level of the mercury flow transmission coefficient C was known to no better than ±10 percent. The major uncertainty was the temperature of the liquid mercury. This uncertainty was caused by normal error of the thermocouple readings and possible temperature gradient between the thermocouple and liquid mercury.

Figure 4 is a photograph of the experimental vaporizer (sample 1 through 9) showing the porous plug, heater, and thermocouple lead. The iron-constantan thermocouple was spot welded 0.35 cm from the end of the tube and the lead wires were wrapped half way around the tube to minimize lead conduction losses. The thermocouple output was measured by a calibrated recorder. The heater was wrapped several times around the tube with the last coil 0.5 cm from the end of the tube. Approximately 6 watts were needed to heat the vaporizer to 300° C. Samples 10 and 11 were prototype SERT II hardware and had slightly different heater construction and thermocouple placement. The remaining samples, 12 through 17 were not heated during tests.

Nitrogen Flow Determination

Figure 5 is a schematic drawing of the apparatus used to measure the flow of nitrogen through samples 1 to 10. Each sample vaporizer tube was placed in a rubber stopper and the rubber stopper sealed a passage between two volumes. The upper volume was charged, stepwise with nitrogen, over a pressure range of 0.1 to 10 N/cm^2 (8 to 800 torr). The lower volume was evacuated to about 5×10^{-2} torr by a mechanical pump and then valved off. As gas flowed through the porous sample, the rate of the pressure rise in the lower volume was measured and, knowing the volume, the flow rate could be computed. This procedure was repeated for each step-change in pressure of the upper volume.

The upper volume was sufficiently large that its pressure remained effectively constant during a datum point. The pressure in the lower volume was small enough that the back flow due to this pressure was either neglected or included as a minor correction. The reproducibility of data points with this apparatus was ±1 percent for each point. The uncertainty in C at the highest pressure tested (10 N/cm^2) was ±1 percent, becoming inversely larger at lower pressures. See the Results and Discussion for an inconsistency between tests.

Burst Tests

After all mercury and nitrogen flow tests were finished, the experimental vaporizers were hydraulically pressurized to their burst point to determine their stress rupture limit. Additional samples, 12 to 16, were fabricated to obtain stress rupture values for larger diameter and thinner plug samples. Sample 12 was prepared from the same slab of material as was sample 1, but the plug thickness ℓ was reduced to half by EDM before welding in its tube. The rupture stress was calculated using Eq. (4) with a k value of 0.75 and values of D, ℓ, and burst pressure listed in table II. Two gages, each ±3 percent of full scale, were used to measure the burst pressure. One gage 2800 N/cm^2 (4000 psig) was used for pressures under 2800 N/cm^2, and the other gage 10 000 N/cm^2 (15 000 psig) was used for higher pressures. Water continually leaked through the porous samples, but the capacity of the hydraulic pump was sufficient to overcome this leak and still build up pressure to the burst point.

Results and Discussion

Mercury Vapor Flow

The experimentally determined value of mercury vapor transmission coefficients as a function of upstream pressure are plotted in Fig. 6 for eleven samples tested. The results are generally as expected, namely, the magnitude of C changes between samples in a predictable fashion based on powder size (pore diameter) and density (sintering

temperature and time) as listed in table I. For example, samples 1 to 4 of the same powder size, but increasing sintering, show progressively lower values of C as the sintering increases. The comparison between samples of different powder sizes is not direct because sintering conditions were also changed as well.

Most samples were tested over a range of vapor pressures above 2.5 N/cm^2 because at normal thruster operation the vaporizer, which is close-thermally coupled to the thruster, is at an equilibrium temperature corresponding to that vapor pressure or greater. Many samples at 2.5 N/cm^2 were at a high enough pressure that flow was no longer free molecular and hence C was not constant with pressure. The variation of C with pressure was similar for all samples and also agreed within ±20 percent of the free molecular C variation obtained with nitrogen flow. (Nitrogen flow results are discussed in the next section.) For some tests, such as sample 6, the vapor pressure was decreased below 2 N/cm^2. For these tests a level value of C was obtained corresponding to free molecular flow in the pores.

Where obtained, a vapor pressure "knee" value was used to compute a mean free path length. This path length, as theory predicts,(7) corresponded roughly to the manufacturer's stated pore diameter. This correspondence, however, was not as good as that between the stated pore diameter and the measured intrusion level. Perhaps the lack of correspondence is due to the somewhat arbitrary faring of the curves to obtain a "knee." More data points could remove this arbitrariness, but the taking of such data points would be more time consuming than a direct measure of the intrusion level.

In summary, the mercury flow tests show that hot mercury vapor flows with normally predicted behavior over the range tested. This behavior permits design to other flow levels or porous materials if the value of C is known. Also, as the variation of C with vapor pressure (temperature) is regular and reproducible, the variation will cause no difficulty when the vaporizer is used in a closed-loop thruster control system. Although none of the tests of this investigation were long term (thousands of hours), the 50 000 hours of accumulative vaporizer operation during the SERT II program without any noticeable change in C indicate an invariance of C with operating time.

Nitrogen Flow

The purpose of flowing nitrogen gas through the vaporizer samples was to determine if, in the future, some gas such as nitrogen could be used as a substitute for the more time-consuming mercury vapor flow calibration. Figure 7 is a plot of transmission coefficient C versus upstream pressure for the samples tested. Table II lists the value of C for the horizontal portion of each sample.

The data of Fig. 7 exhibited a constant (horizontal) value of C in the low pressure or free molecule flow range. At higher pressures the value of C increased with pressure. The "knee value" of each curve occurred at a pressure of three to five times less than the "knee value" for the corresponding curve when tested with mercury. Assuming that the "knee value" occurs when the free molecular path equals the pore diameter, there should be a factor of 4 to 5 less, because of a 2:1 temperature difference and a molecular size difference.(17) The nitrogen "knee values" of Fig. 7, when used to estimate a pore diameter and a liquid mercury intrusion value by means of Eq. (2), resulted in more uncertainty than if the intrusion value were estimated directly by using the manufacturer's stated pore diameter.

The measured nitrogen free molecular flow value of C was somewhat inconsistent with the value reported by the manufacturer. For most tests (samples 2 to 5, 7) the measured C value was 5 to 20 percent higher, but two tests, samples 9 and 1, were equal and lower, respectively, than the manufacturer's value. In two other tests, samples 6 and 8, the measured value was 30 and 40 percent higher, respectively. The minor discrepancies (less than 20 percent) could be caused by differences in measurement technique or sample preparation. The larger discrepancy was believed caused by an intermittent leak in a bypass valve between the upper and lower volumes. Unfortunately, the samples were destroyed in burst testing before the valve leak was discovered.

In summary, the nitrogen tests indicated that nitrogen flowed through the porous tungsten in a manner similar to mercury vapor. There was general agreement in the values of C measured and in the break (knee value) from free molecular to slip flow. However, the agreement was inconsistent enough that mercury flow calibrations must still be made for accurate or flight vaporizer use. If a research vaporizer is suspected of a gross flow change (more than 20 percent), a nitrogen flow test offers a convenient and short procedure to check for that change.

Liquid Mercury Intrusion

The major motivation in preparing the porous material of samples 1 to 9 was to extend the liquid mercury pressure intrusion level without sacrificing flow (maintaining high C values) or fabrication ease (maintaining high density for good welding). To this extent the results were disappointing. The anticipated gain in intrusion pressure level was double that of earlier porous material, but the actual increase in intrusion level was only about 15 percent over the SERT II neutralizer vaporizer material (sample 11). The actual intrusion level at room temperature for each sample tested is listed in table II. The highest level measured, 108 N/cm^2 (157 psia), was for sample 4. Figure 8 is a plot of the measured intrusion pressure value in table II versus a calculated value based on the pore diameter of table I and the use of Eq. (2). Samples 10 and 11 are close to the average value each for over 20 vaporizers which were tested during the SERT II program. Most of measured values of intrusion pressure are lower than the calculated values, probably due to a few pores larger than the average values presented in table I.

The predominate exceptions to predicted intrusion level were: (1) sample 8 too low, not plotted because of an extremely low intrusion level caused by a hair line crack, roughly parallel to the circumferential weld and extending around one-fourth of the circumference; (2) sample 11 too high, for

which doubt exists in the manufacterer's stated pore diameter. Using an estimated pore size based on a comparison of pore sizes for other powder sizes and sintering conditions, a calculated intrusion pressure level about 10 percent higher than the measured value was obtained for sample 11; (3) samples 7 and 9 too low, the simplest explanation being random large pores which permitted early penetration. This explanation was supported by the observed intrusion pattern of liquid mercury drops on the downstream face of the plug. The pattern was not randomly uniform, but rather the mercury drops appeared only at several consistent spots. With the exception of sample 8 no hairline cracks were observed in any sample using X60 power examination.

Three samples were tested for liquid mercury intrusion pressure level while at operating temperature (300° C). Each sample tested showed a lower intrusion pressure level when hot. Samples 2 and 3 were each 25 percent lower, while sample 4 was 3 percent lower than the level measured at room temperature. The hot intrusion pressure level is of less practical interest because the maximum liquid pressure level will generally be experienced during launch vibration when the thruster system is near room temperature.

Of more practical interest is any change in flow rate or C as a function of liquid pressure because the liquid feed pressure may change during the life of a mission. Values of C were therefore measured with stepwise increase in liquid pressure. As a measure of any change, a sensitivity factor was defined as the ratio at constant temperature of C at 51 N/cm^2 pressure to C at 17 N/cm^2 pressure. This ratio was measured for each sample and is listed in table II as the liquid pressure sensitivity. The values of this ratio are small and seem to be random with respect to porous structure. Any flight feed system should be designed with a liquid feed pressure well under 51 N/cm^2 (75 psia) and the 10 percent variation of this ratio can be compensated by a small (<10° C) change in a closed-loop vaporizer operating temperature.

Of great practical interest is what happens to a vaporizer if it should be intruded with liquid mercury. After mercury is intruded into the porous tungsten vaporizer, liquid flow will continue through the pores until the liquid pressure is reduced. After the liquid flow once stops, three plug flow characteristics have been observed to exist: (1) liquid will flow at low pressures low as 1/10 of the original intrusion level); (2) liquid will not flow until the original intrusion level is again applied, but the vapor transmission coefficient C has been increased, up to a factor of 10 higher; and (3) complete return to both normal intrusion pressure level and vapor flow. The main vaporizers of SERT II (sample 10) exhibited characteristics (1) or (2) in about 90 percent of the cases tested, but the record of the neutralizer vaporizer (sample 11) was better (about 50 percent). Characteristics (1) or (2) were both considered to be vaporizer failures with SERT II program. The new porous material of this study exhibited characteristic (3) in six (75 percent) samples, 1, 3, 5, 6, 7, and 9. Table II lists values of C for before and after mercury intrusion. In two samples, 2 and 4, the value of C was increased by mercury intrusion to a level 11 and 4 times higher, respectively. Such increases in C are beyond the limit of the flow range of a closed-loop thruster vaporizer. These two samples, however, did return to completely normal characteristics after a 400° C vacuum bakeout. One sample, sample 8, exhibited characteristic (1), and this was attributable to the previously noted hairline crack in the weld-porous material interface area.

Many laboratory vaporizers that exhibited characteristic (1) or (2) after inadvertent mercury intrusion have been restored to service by a 400° C vacuum bakeout. To the author's knowledge, the bakeout procedure has never failed to restore to normal a vaporizer which had failed due to mercury intrusion. Mechanical damage (cracking) or contaminate wetting[12] has caused the only permanent failures. The reason for a given flow characteristic probably lies either in the sintering and powder used in fabrication, or in the electron-beam welding method applied. The exact specification to obtain characteristic (3) remains to be determined in the future.

Burst Tests

Table II lists the liquid burst pressure measured for 13 samples and the rupture stress calculated using Eq. (4) and a k value of 0.75. The general level of rupture stress, 16 000 to 20 000 N/cm^2 (23 000 to 29 000 psi) was that estimated by the porous material manufacturer. There is a dependence between rupture stress and sintering time. Samples 2 and 4 with progressively longer sintering times also had progressively higher rupture stress values. The larger diameter plugs had the same general level of rupture stress as did the normal, 0.59-cm diameter samples. The use of Eq. (4) therefore gives good design correlation for vaporizer plugs of different diameter D or thickness ℓ.

Every burst test result is included in table II. None were excluded. Two samples, 7 and 16, showed lower values of rupture stress. In observing the rupture pattern, samples 7 and 16 had plug fragments missing and a crack pattern emanating from an area near the edge of the plug as if the rupture began there at a flaw. The other samples had fragments missing from the center and a series of near radial cracks as if the failure occurred in the center.

If the porous plug were of uniform composition and strength, the burst failure for a simply-supported edge would be expected at the center where the highest stress value occurs. Therefore, the calculated values of rupture stress listed in table II may be pessimistically low because of an arbitrary selection of a k value for a "clamped edge" support. If the plug is simply-supported, the calculated stress rupture values would be 65 percent higher. The difference in calculated stress rupture values is only of interest should a differently supported plug be made. In that case, a burst test would be required to determine a maximum working pressure.

One sample, sample 12, was EDM (Electric Discharge Machined) to half thickness to see if slab material could be made thinner to increase flow and yet maintain its strength. As the stress rupture for sample 12 was normal, it can be assumed that

flow increases can be obtained by EDM thinner material. After EDM, however, the surface pores must be opened by treatment such as used in Ref. 6 to prevent flow restriction.

Collimated Hole Sample

Figure 9 shows two successive enlargements of sample 17, which is called a "collimated hole" structure by its manufacturer. It is made of stainless steel and consists of near-parallel, small straight capillary tubes extending through the material. The holes are roughly circular with a diameter of 13 microns. The thickness ℓ of the slab is 0.050 cm and the hole open fraction is 0.27. There are also thick hexagonal webs, presumably for support, that make a large overall honeycomb pattern.

The major concern for the eventual use of this type of material for a vaporizer is its ability to withstand liquid mercury intrusion. This value was 9.7 N/cm^2 (730 torr), 27 percent less than theoretical based on Eq. (3). In addition, the penetrated mercury completely flowed out of the pores when the intrusion pressure was reduced, so this material has flow characteristic (3), complete recovery of its normal intrusion pressure and vapor flow rate. As the flow capacity of this sample exceeded the measuring capability of either the mercury or nitrogen flow apparatus, its flow was not directly measured. The porous structure, however, is very regular and similar to the ideal structure of Ref. 5, so a calculated value of transmission coefficient C should agree closely with measured values. The calculated value of C for this sample was 0.01. Because of the high flow of this material, its use will probably be limited to very large (1.5-m diam) thrusters. Its relatively low intrusion pressure will present design problems for its use in flight thrusters.

Design and Fabrication Recommendations

Based on the results of this test program and the experience gained in the SERT II flight development, the following vaporizer procedures should be followed in any future flight program. Porous tungsten vaporizers should be fabricated under clean conditions to avoid contaminants that might cause surface wetting and to close mechanical tolerances to avoid welding cracks. A final fabrication step may be a 1-hour air back at 200° C to insure a good surface oxide film which prevents surface wetting. Finished vaporizers should be flow calibrated with mercury vapor and checked for proper liquid mercury intrusion pressure level. After mercury intrusion a 400° C vacuum bake insures removal of any trapped liquid mercury. Finished vaporizers should also be pressure checked to one-half of their calculated burst pressure to assure that there are no flaws which would cause premature stress cracking.

A design problem remains to protect the vaporizer from mercury intrusion during launch vibration. Porous material technology may at best double the intrusion pressure levels found by this report, but future flight thruster arrays could have dynamic pressure peaks ten times higher than existing intrusion pressures. The vibration intrusion problem could be solved by placing a valve in the mercury line between the tank and each vaporizer. Such a valve has not yet been tested nor developed for flight use in a mercury feed system. Another approach is to construct a "surge volume" in the liquid mercury line. This surge volume which could be a bellows and spring would dampen the dynamic pressure peaks before they increase to the intrusion level. The "volume" could also be lateral, blind-end, pores of a diameter larger than the vaporizer pores.(18) If vaporizers had flow characteristic (3), complete recovery after intrusion, it would be possible to accept the small amount of mercury forced through during dynamic vibration peaks and assume this mercury will evaporate during thruster preheat. The recommended design approach, however, is to use a propellant line valve or surge volume to insure trouble-free vaporizer operation. Flow characteristic (3) nevertheless is valuable insurance and can be verified during vaporizer calibration.

Concluding Remarks

The design and fabrication sections and the tests performed herein form the basis for confident use of porous tungsten vaporizers for future large-size Kaufman thrusters and thruster array propellant feed systems. Each flight vaporizer can be pretested with mercury to obtain or check flow rate, liquid mercury intrusion pressure, and mechanical strength without harm or change. Nitrogen gas may be used when an approximate flow calibration is needed. A complete normal return after liquid mercury intrusion, was observed in a number of samples and is a desirable property should a vaporizer be accidentally intruded with mercury. But this characteristic should not be depended upon to circumvent the dynamic launch line pressure problem. When long propellant lines are required, a positive way, such as a valve or surge volume should be used to prevent high dynamic pressures at the vaporizer plug. The successful welding of larger diameter plugs permits high flow vaporizers to operate at low vapor pressure where lighter-weight feed systems can be designed. The high values of stress rupture measured will also permit the design of thinner plugs if more flow is needed. Other porous vaporizer materials were briefly considered but offered little or no improvement over the use of porous tungsten. The high past success with porous tungsten and the material technology already developed, strongly recommend its continued use in future thruster systems.

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TABLE I. - MANUFACTURER'S CHARACTERISTICS OF POROUS MATERIAL

Sample Number	Manufacturer batch number	C Transmission coefficient	Pore diameter μ	Powder diameter μ	Slab density, fraction of theoretical	Slab thickness, cm	Sintering	
							Temp. °C	Time min
1, 12	1	2.1×10^{-4}	1.9	4.2	0.615	0.144	1850	42
2, 15, 16	2A	0.81×10^{-4}	1.9	4.2	0.731	0.140	2100	75
3	2D	0.32×10^{-4}	1.8	4.2	0.798	0.135	2100	195
4	2E	0.18×10^{-4}	1.6	4.2	0.813	0.139	2100	240
5, 13, 14	3A	2.1×10^{-4}	1.8	4.2	0.683	0.084	2000	90
6	3B	1.0×10^{-4}	1.6	4.2	0.746	0.082	2100	75
7	4	0.48×10^{-4}	1.3	3.5[a]	0.762	0.142	1950	60
8	5A	0.88×10^{-4}	1.2	3.5[a]	0.766	0.077	1990	60
9	5B	1.48×10^{-4}	1.3	3.5[a]	0.709	0.079	1875	60
10	5433	1.9×10^{-4}	2.4	6.2[a]	0.695	0.142	2000	120
11	5207	0.58×10^{-4}	2.4	3.5	0.760	0.130	2100	300
17	CHS	100×10^{-4}	13	---	0.27	0.050	----	---

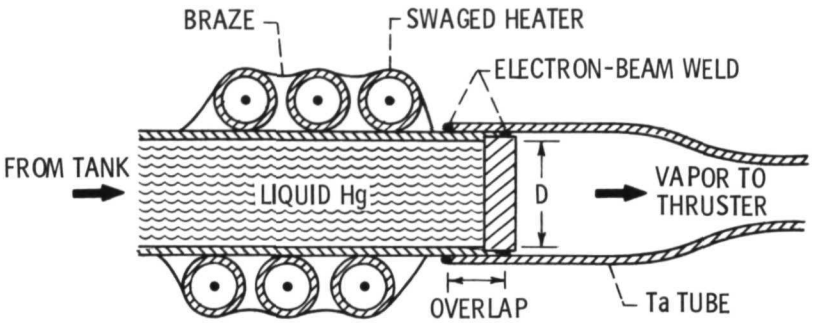
[a]Spherical Powder (other samples are made of Angular Powder)

Note: Sample 10 was SERT II Main Vaporizer S/N-22
Sample 11 was SERT II Neutralizer Vaporizer P-1

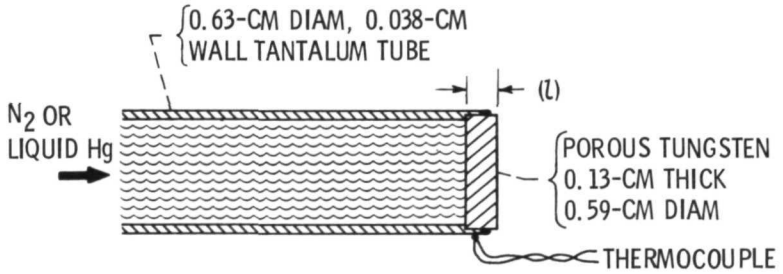
TABLE II. - TEST RESULTS

Sample number	Transmission coefficient, C					Liquid pressure sensitivity at 300° C $\frac{C_{51}\ N/cm^2}{C_{17}\ N/cm^2}$	Liquid intrusion pressure at 20° C N/cm^2	Plug diameter D cm	Plug thickness ℓ cm	Liquid burst pressure at 15° C N/cm^2	Rupture stress S N/cm^2
	Nitrogen, 20° C		Mercury, 300° C (3.26 N/cm^2)								
	By manufacturer	Welded in 0.63 cm diameter tube	Initial test	After Hg intrusion	After bakeout						
1	2.24×10^{-4}	2.03×10^{-4}	1.85×10^{-4}	1.90×10^{-4}	1.82×10^{-4}	1.00	89	0.59	0.144	----	------
2	0.81×10^{-4}	0.89×10^{-4}	0.91×10^{-4}	10×10^{-4}	0.95×10^{-4}	0.95	102	0.59	0.140	5100	17×10^{3}
3	0.32×10^{-4}	0.36×10^{-4}	0.25×10^{-4}	0.25×10^{-4}	---------	1.08	99	0.59	0.135	5600	20×10^{3}
4	0.18×10^{-4}	0.19×10^{-4}	0.14×10^{-4}	0.63×10^{-4}	0.14×10^{-4}	0.97	108	0.59	0.139	6200	21×10^{3}
5	2.10×10^{-4}	2.50×10^{-4}	1.93×10^{-4}	1.93×10^{-4}	1.98×10^{-4}	1.02	94	0.59	0.084	1450	14×10^{3}
6	1.01×10^{-4}	1.33×10^{-4}	1.15×10^{-4}	1.42×10^{-4}	1.15×10^{-4}	1.06	95	0.59	0.082	1600	16×10^{3}
7	0.48×10^{-4}	0.56×10^{-4}	0.59×10^{-4}	0.67×10^{-4}	0.60×10^{-4}	0.90	96	0.59	0.142	3300	11×10^{3}
8	0.88×10^{-4}	1.26×10^{-4}	1.03×10^{-4}	---------	1.10×10^{-4}	----	35	0.59	0.077	1600	18×10^{3}
9	1.48×10^{-4}	1.58×10^{-4}	1.47×10^{-4}	1.47×10^{-4}	---------	0.99	103	0.59	0.079	1650	17×10^{3}
10	1.90×10^{-4}	---------	1.59×10^{-4}	---------	1.59×10^{-4}	1.05	62	0.59	0.142	----	------
11	0.58×10^{-4}	---------	0.48×10^{-4}	---------	0.48×10^{-4}	1.05	90	0.27	0.130	----	------
12	---------	---------	---------	---------	---------	----	---	0.59	0.071	1250	16×10^{3}
13	2.10×10^{-4}	---------	---------	---------	---------	----	---	1.20	0.084	520	20×10^{3}
14	2.10×10^{-4}	---------	---------	---------	---------	----	---	1.20	0.084	410	16×10^{3}
15	0.81×10^{-4}	---------	---------	---------	---------	----	---	1.80	0.145	620	18×10^{3}
16	0.81×10^{-4}	---------	---------	---------	---------	----	---	1.80	0.145	240	7×10^{3}
17	1×10^{-2}	---------	---------	---------	---------	----	9.7	1.4	0.051	----	------

Note: 1 N/cm^2 = 1.45 psi

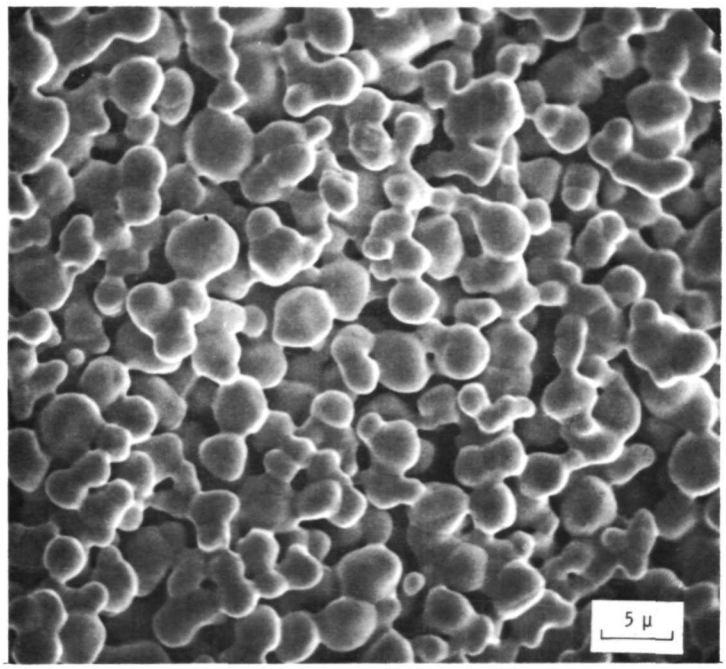


(a) SERT II VAPORIZER TYPE.

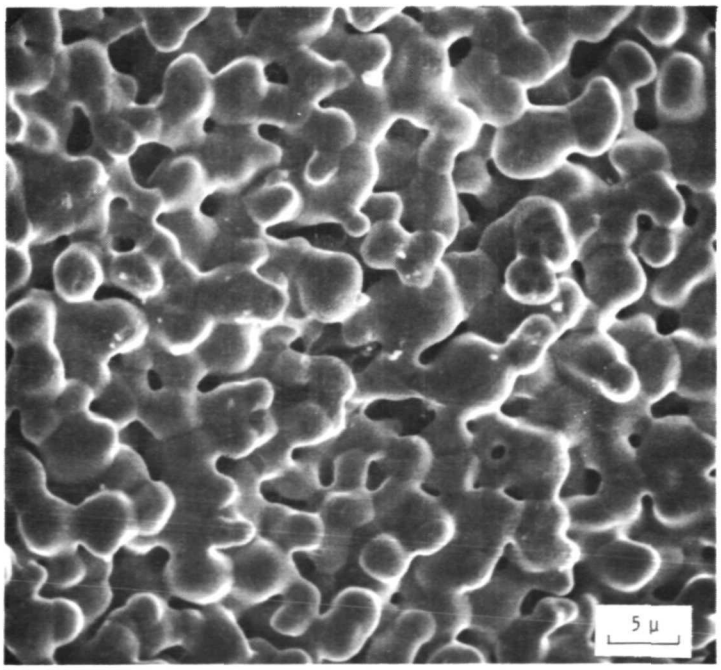


(b) EXPERIMENTAL VAPORIZER.

Figure 1. - Vaporizer cross sections.



(A) SAMPLE 1, LIGHTLY SINTERED.



(B) SAMPLE 2, AVERAGE SINTERING.

Figure 2. - Electron-scan microphotographs.

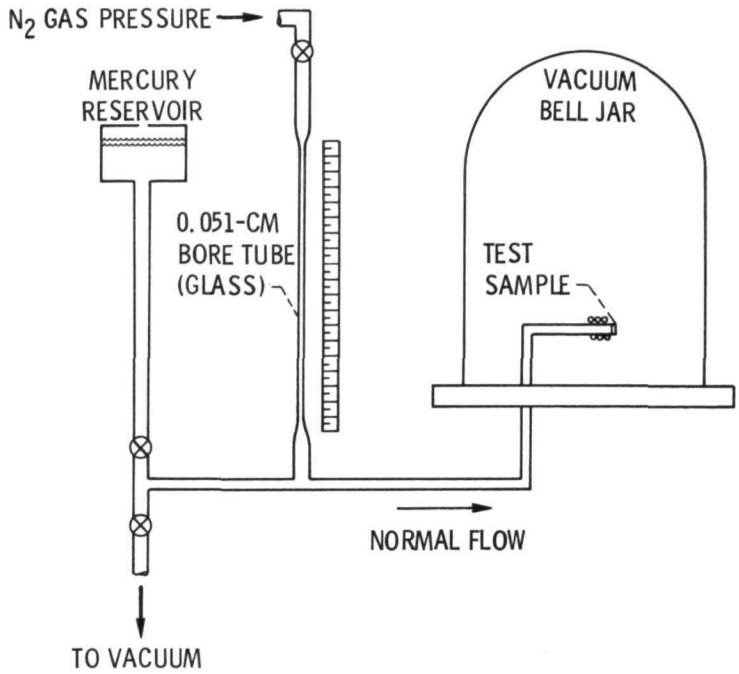


Figure 3. - Mercury test flow apparatus.

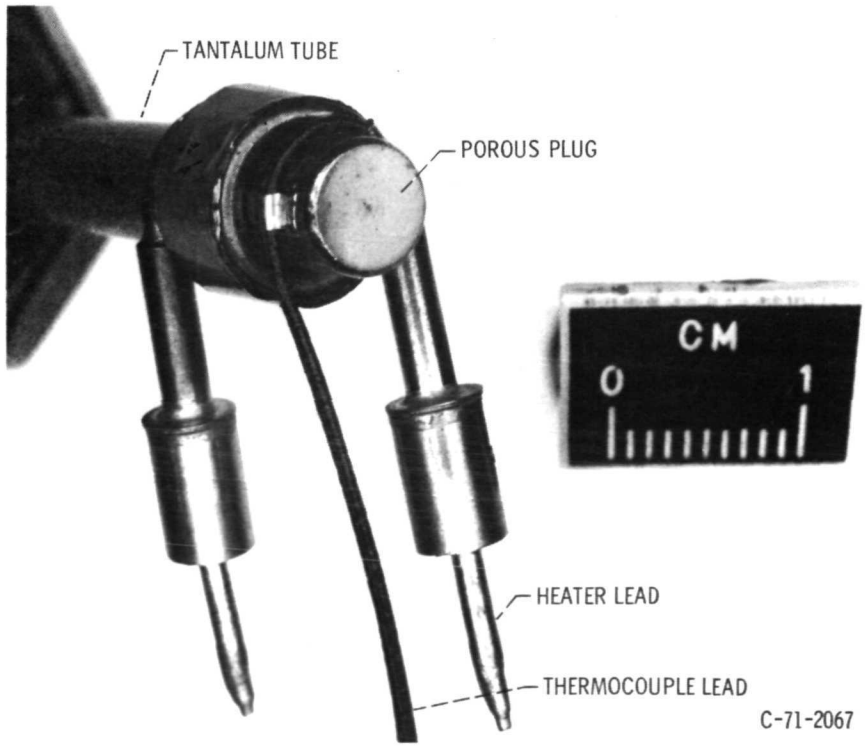


Figure 4. - Experimental vaporizer.

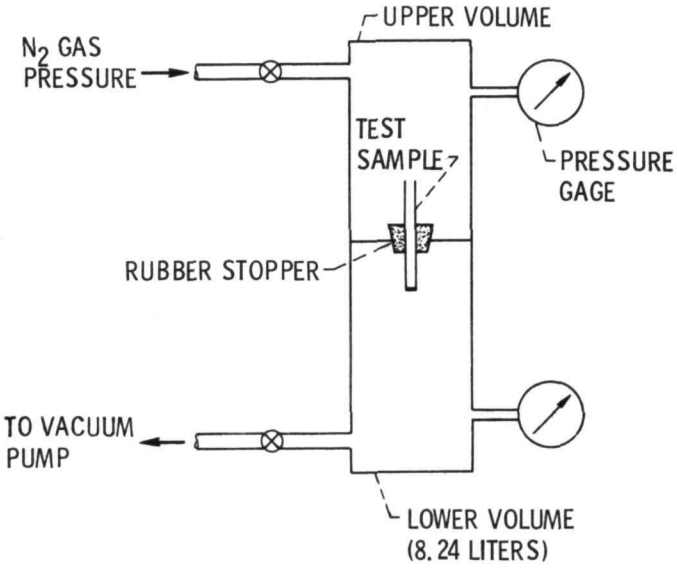


Figure 5. - Nitrogen test flow apparatus.

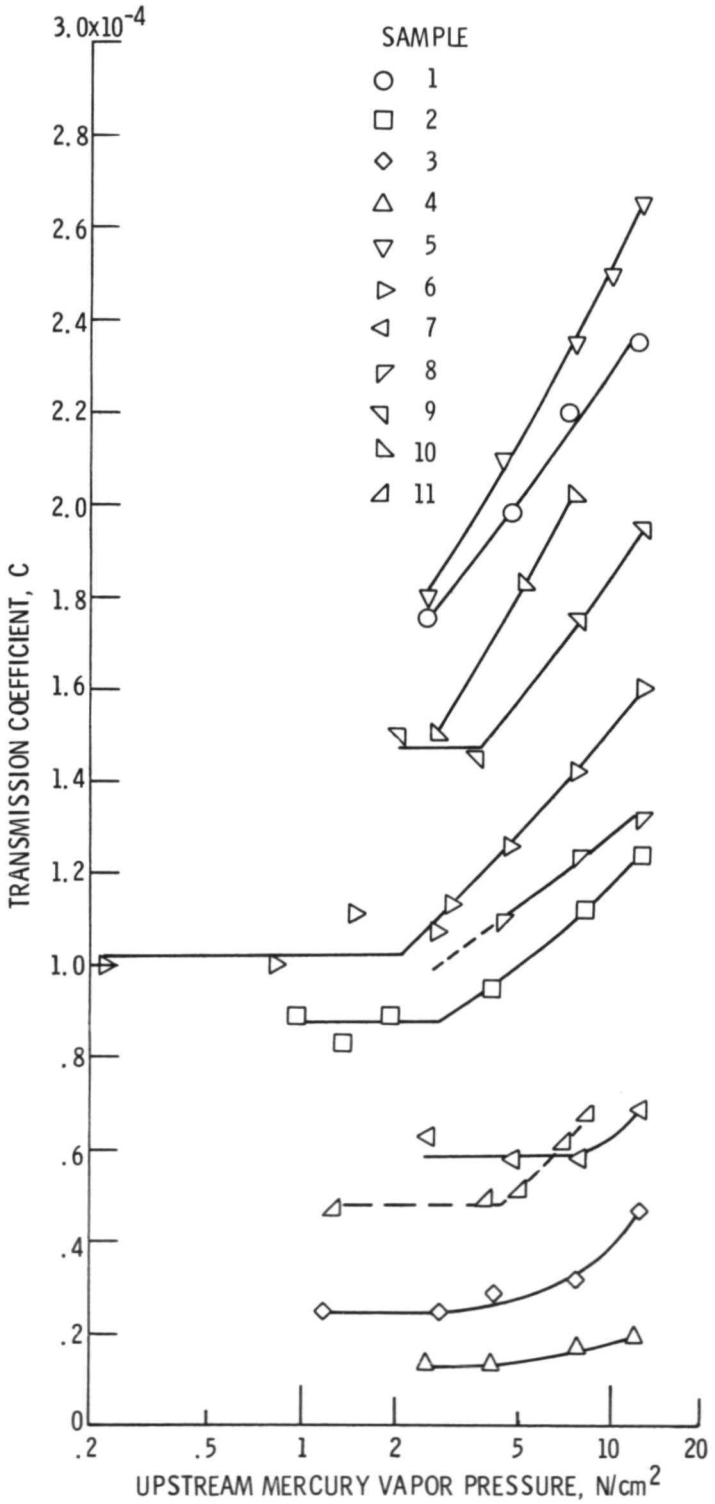


Figure 6. - Mercury flow through porous tungsten at equilibrium temperature - vapor pressure.

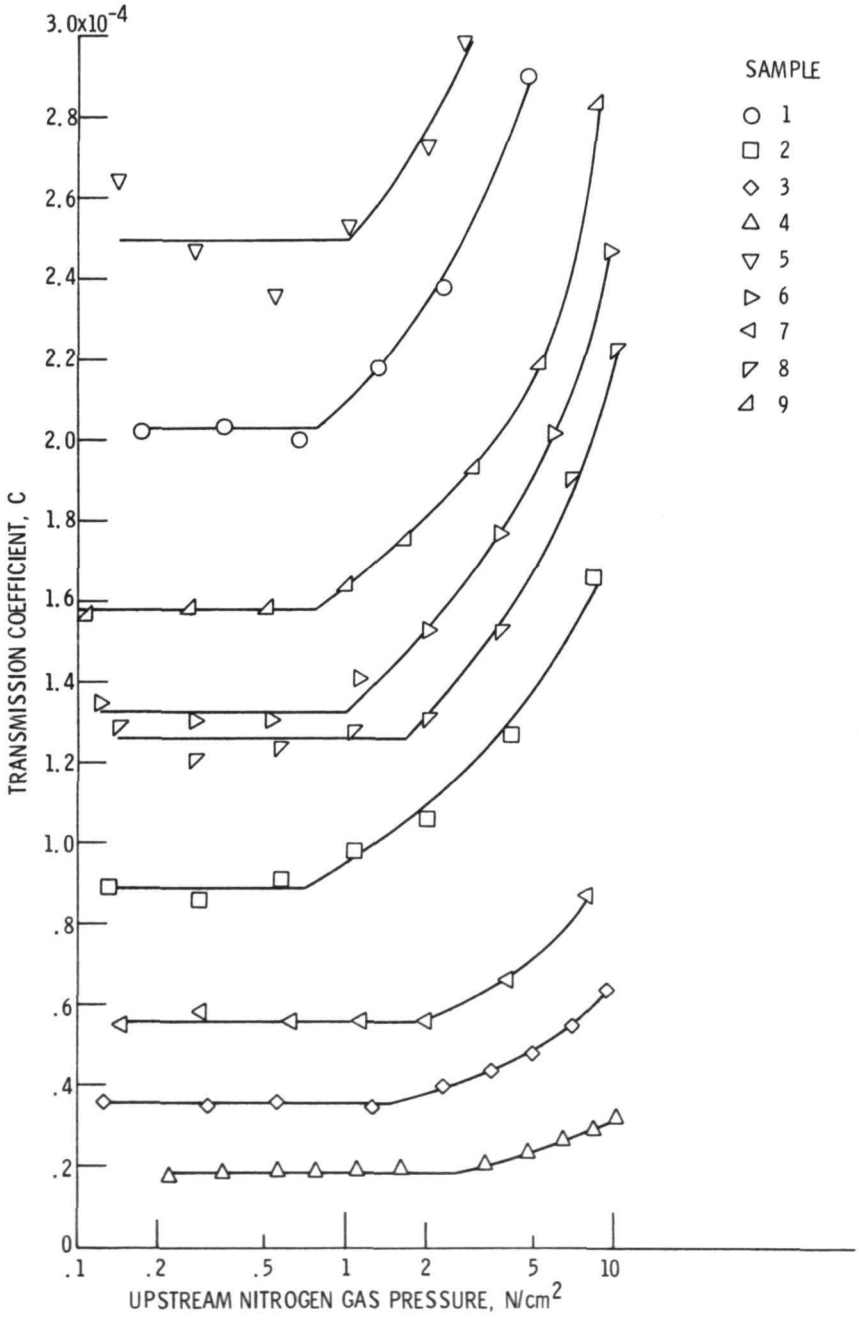


Figure 7. - Nitrogen flow data through porous tungsten at room temperature.

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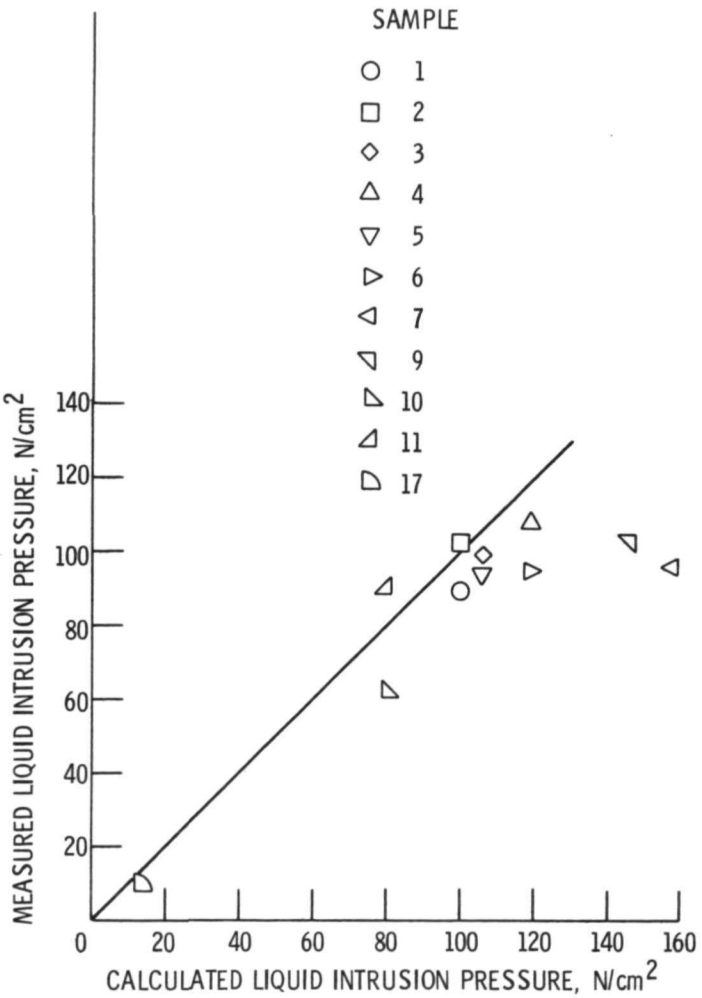
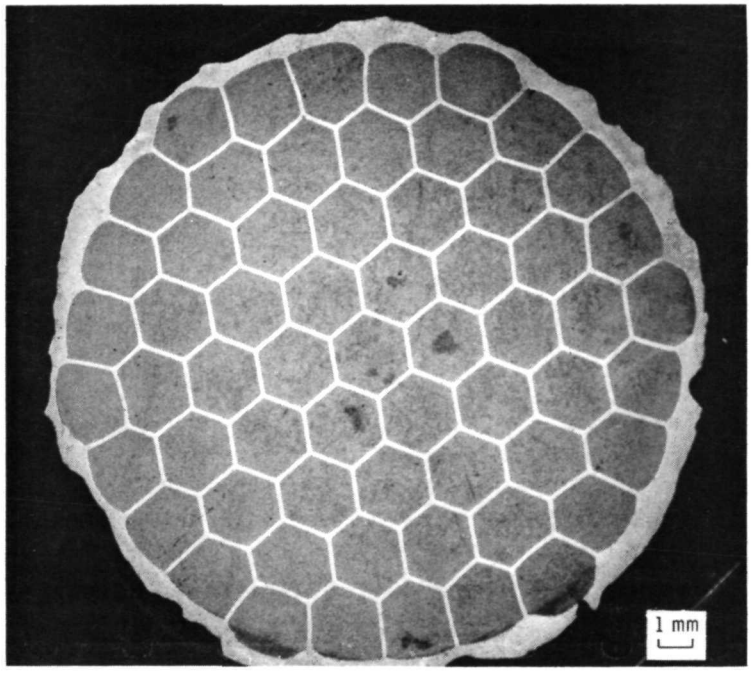
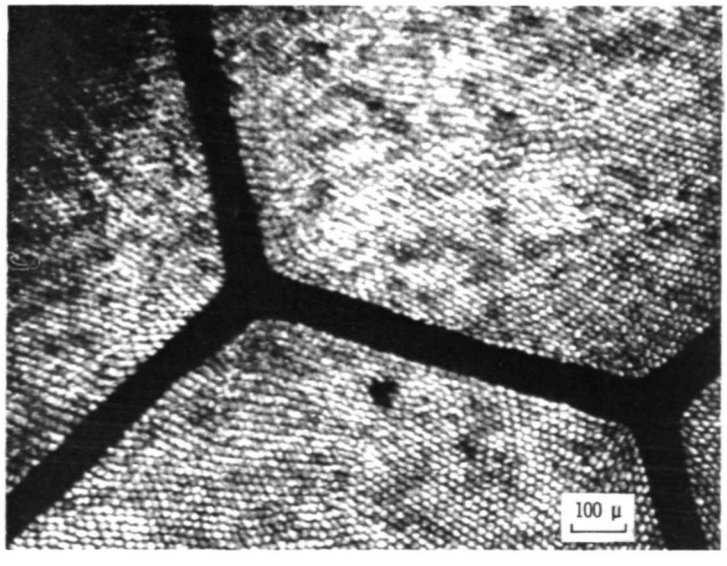


Figure 8. - Comparison of calculated with measured liquid mercury intrusion pressure.



(A) MAGNIFICATION, X4.



(B) MAGNIFICATION, X56.

Figure 9. - Microphotographs of collimated hole, sample 17. (Enlargement, X56).